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(71) Applicant: SUMITOMO ELECTRIC INDUSTRIES, LTD.
5-33, Kitahama 4-chome, Chuo-ku
Osaka-shi, Osaka 541(JP)

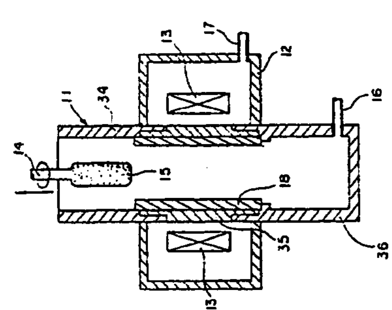
(72) Inventor: Tsuchiya, Ichiro, c/o Yokohama Works Sumitomo
Electric Industries Limited, 1, Taya-cho
Sakae-ku,Yokohama-shi, Kanagawa-ken(JP)
Inventor: Saito, Masahide, c/o Yokohama Works Sumitomo
Electric Industries Limited, 1, Taya-cho
Sakae-ku,Yokohama-shi, Kanagawa-ken(JP)
Inventor: Ishikawa, Shinji, c/o Yokohama Works Sumitomo
Electric Industries Limited, 1, Taya-cho
Sakae-ku,Yokohama-shi, Kanagawa-ken(JP)
Inventor: Ohga, Yuichi, c/o Yokohama Works Sumitomo
Electric Industries Limited, 1, Taya-cho
Sakae-ku,Yokohama-shi, Kanagawa-ken(JP)
Inventor: Kanamori, Hiroo, c/o Yokohama Works Sumitomo
Electric Industries Limited, 1, Taya-cho
Sakae-ku,Yokohama-shi, Kanagawa-ken(JP)

(74) Representative: Brauns, Hans-Adolf, Dr. rer. nat. et al
Hoffmann, Eitle & Partner, Patentanwälte
Arabellastrasse 4
W-8000 München 81(DE)

(54) **Furnace for production of optical fiber preform.**

(57) There is provided a furnace comprising a muffle tube made of a gas impermeable and heat resistant material, a heater in a furnace body which surrounds the muffle tube and an inner cylinder made of a heat resistant material which is detachably fitted inside the muffle tube near the heater for a thermal treatment of a quartz glass preform by inserting the preform in the muffle tube, wherein said muffle tube comprises a material selected from a group consisting of highly pure carbon coated with gas impermeable silicon carbide, sintered silicon carbide coated with gas impermeable silicon carbide, gas impermeable silicon carbide and highly pure carbon coated with gas impermeable carbon, and said inner cylinder comprises a material selected from a group consisting of highly pure carbon, highly pure carbon coated with gas impermeable silicon carbide and highly pure carbon coated with gas impermeable carbon.

Fig. 2



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FURNACE FOR PRODUCTION OF OPTICAL FIBER PREFORM

BACKGROUND OF THE INVENTION

Field of the invention

The present invention relates to a furnace in which a porous glass preform for an optical fiber preform is thermally treated, for example, dehydrated, doped or sintered to produce a highly pure quartz glass preform for an optical fiber.

Description of the Related Art

In the production of an optical fiber preform, a porous glass preform consisting of fine quartz glass particles produced by the Vapor Phase Axial Deposition or the Outside Chemical Vapor Deposition is dehydrated, densified and vitrified. During the dehydration, the densification and/or the vitrification, fluorine is added to the glass to control a refractive index profile of the preform. A heating furnace comprising a muffle tube is used for the dehydration, the densification or the vitrification.

One example of such heating furnace comprising the muffle tube is shown in Fig. 1. The conventional heating furnace comprises a hollow furnace body 12 and a muffle tube 11 which passes through the furnace body 12. A heating member 13 is placed in the furnace body 12 which has an inlet 17 for introducing an inert gas into the furnace body in order to prevent deterioration of the muffle tube, and the muffle tube 11 has an inlet 16 for introducing an atmosphere gas such as Cl_2, SiF_4 or He into the muffle tube. In the use of this heating furnace, a porous preform 15 is thermally treated by means of the heater 13 while it is suspended from a supporting rod 14.

It is known that the muffle tube is made of highly pure carbon coated with silicon carbide (hereinafter referred to as SiC).

For example, in Japanese Patent Kokai Publication No. 201634/1986, a carbon made muffle tube coated with SiC is used, and a surface of the SiC coating is further oxidized.

Since such muffle tube comprises carbon as a substrate, it can be kept at a high temperature without deterioration, whereby it can be heated and cooled many times with careful temperature change. In addition, the carbon substrate is hardly oxidized since it is coated with SiC so that impurities contained in the carbon substrate do not penetrate the glass preform. Because the surface of the SiC coating has been oxidized, the muffle tube does not deteriorated with Cl_2 or SiF_4.

As described above, the conventional muffle tube comprises the carbon made substrate a surface of which is coated with SiC and the surface of the SiC coating is oxidized.

However, it is known that there arise various difficulties during the fabrication of such muffle tube. Firstly, conditions of the SiC coating oxidation are so sensitive that it is difficult to uniformly oxidize the SiC coating. As a result, some portions are not oxidized and/or the coating peels off due to internal stress within the oxidized SiC coating.

The oxidation of the SiC coating is carried out in an atmosphere comprising oxygen during the dehydration or the fluorine addition of the glass preform. Alternatively, the muffle tube is baked in an oxygen atmosphere. If the SiC coating partially peels off, the carbon made substrate of the muffle tube is severely oxidized during the oxidation step of the SiC coating, whereby a life of the muffle tube is extremely reduced.

As described above, there are severe problems during the oxidation of the SiC coating. The SiC coating is rapidly deteriorated with the fluorine-containing gas when the oxidation of the SiC coating is omitted. Such deterioration begins at a temperature of 1200 °C with SiF_4 gas, and rapidly proceeds at a temperature above 1400 °C. As a result, the carbon made substrate under the SiC coating is oxidized by a small amount of oxygen, whereby the life of the muffle tube is extremely shortened.

The glass preform may be densified/vetrified in an atmosphere consisting of an inert gas depending on a kind of an optical fiber which is drawn from the preform. In such case, a surface of the SiC coating is not required to be oxidized for the protection from Cl_2 or SiF_4, and then it is rather preferable to omit the oxidation step since the sensitive conditions are required. However, even in such case, the SiC coating may be oxidized with a small amount of oxygen in the muffle tube.

SUMMARY OF THE INVENTION

It is an object of the present invention to provide a furnace in which porous glass preforms are thermally treated for a long period of time to produce highly pure quartz glass preforms for an optical fiber without decrease of strength of the optical fiber.

In the first aspect of the present invention, there is provided a furnace comprising a muffle tube made of a gas impermeable and heat resistant material, a heater in a furnace body which surrounds the muffle tube and an inner cylinder made of a heat resistant material which is detachably fitted inside the muffle tube near the heater for a thermal treatment of a quartz glass preform by inserting the preform in the muffle tube, wherein said muffle tube comprises a material selected from a group consisting of highly pure carbon coated with gas impermeable silicon carbide, sintered silicon carbide coated with gas impermeable silicon carbide, gas impermeable silicon carbide and highly pure carbon coated with gas impermeable carbon, and said inner cylinder comprises a material selected from a group consisting of highly pure carbon, highly pure carbon coated with gas impermeable silicon carbide and highly pure carbon coated with gas impermeable carbon.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 schematically shows a cross-sectional configuration of a conventional heating furnace, and

Fig. 2 schematically shows a cross-sectional configuration of one embodiment of the present furnace.

DETAILED DESCRIPTION OF THE INVENTION

The porous glass preform made of quartz is thermally treated with the heater in the muffle tube in the furnace body. During such treatment, a small amount of oxygen and/or water in the muffle tube firstly reacts with a material of the inner cylinder which is fitted inside the muffle tube and near the heater. Therefore, the water and the oxygen do not directly react with the muffle tube to deteriorate it.

The present invention will be, hereinafter, explained in detail with reference to the accompany drawings. Members which are substantially the same as those of the conventional furnace are given the same numerical numbers as in the conventional furnace, and detailed explanations which are the same as those on the conventional furnace are neglected.

In the embodiment shown in Fig. 2, an inner cylinder 18 is detachably fitted inside a muffle tube 11 near a heater 13. In this embodiment, the muffle tube 11 is made of highly pure carbon coated with gas impermeable SiC. The muffle tube may be made of sintered silicon carbide coated with gas impermeable SiC, gas impermeable SiC or highly pure carbon coated with gas impermeable carbon such as pyrolitic carbon or vitreous carbon. In the case where the SiC or the carbon coating is applied, at least an inner surface of the muffle tube is coated with SiC or carbon. Both surfaces, of course, may be coated with SiC or carbon.

Although the inner cylinder 18 is made of highly pure carbon in this embodiment, at least an inner surface of the inner cylinder may be coated with gas impermeable carbon or SiC.

The porous glass preform 15 is thermally treated, for example, dehydrated, fluorine-added and/or sintered with the heater 13 in an atmosphere comprising a gas such as $SiCl_4$, SiF_4 or He supplied through an inlet 16 in the muffle tube 11. During the treatment, a trace amount of oxygen and water adsorbed in the quartz made porous glass preform 15 is liberated into the atmosphere in the muffle tube 11.

In the case where the conventional furnace without the inner cylinder 18 is used, the following reaction proceeds to deteriorate the SiC coating:

$SiC + 3SiF_4 + 1/2O_2 \rightarrow 4SiF3 + CO$ (1)

Even in the case where only the inert gas is used, the surface of the SiC coating is oxidized with oxygen in the muffle tube according to the following equation:

$2SiC + 3O_2\ 2SiO_2 + 2CO$ (2)

and the SiC coating may be destroyed in the worst case.

However, according to the present invention, the small amount of oxygen which reacts according to the above equation (1) or (2) firstly reacts with highly pure carbon of the inner cylinder according to the following equation (3) to be removed:

$C + 1/2O_2 \rightarrow CO$ (3)

Thus, the deterioration does not reach the muffle tube and the muffle tube is protected.

In addition, in the case where the highly pure carbon made inner cylinder 18 is coated with gas

impermeable carbon or SiC, the atmosphere gas cannot penetrate the inner cylinder 18 itself during the initial term of the use, whereby the protection with the inner cylinder 18 for the SiC coating on the muffle tube 11 is further promoted.

Since the deterioration of the SiC coating according to the equation (1) is remarkable at a temperature above 1200 °C, the inner cylinder is installed so that it covers a portion of an inner surface of the muffle tube near the heater 13 which is heated to a temperature above 1200 °C.

The inner cylinder 18 gradually wears according to the equation (3) during each thermal treatment of the porous preform 15. With such wear, the muffle tube 11 and the SiC coating thereon is fully protected by means of the inner cylinder 18. Although the inner cylinder has to be replaced with a new one depending on an extent of the wear, cost reduction over replacement of the muffle tube can be realized since the inner cylinder 18 is smaller and easily fabricated in comparison with the muffle tube 11.

Example 1

Now, experimental results will be described in below. Quartz made porous glass preforms 15 were thermally treated with using the conventional furnace as shown in Fig. 1 and the present furnace as shown in Fig. 2. Then, pure silica core single mode optical fibers were drawn from the treated preforms.

The used muffle tube 11 in each furnace was made of highly pure carbon coated with SiC, and the inner cylinder 18 was made of highly pure carbon. Temperature conditions and atmosphere gas conditions of the treatments are shown in following Table 1:

Table 1

Treatment	Temperature	Atmosphere gas
Dehydration	950-1100 °C	$SiCl_4$, He
F-addition	1250-1400 °C	SiF_4, He
Sintering	1550-1650 °C	He

After the thermal treatments under the conditions as above with using the furnace as shown in Fig. 1, the SiC coating inside the muffle tube 11 changed its color to black in a portion which was heated to a temperature above 1200 °C, and more than 70 % of the SiC coating disappeared so that the carbon made substrate was exposed and oxidation of a portion of the exposed carbon substrate started. It was expected from these results that at most 250 quartz made porous glass preforms could be thermally treated with a single conventional muffle tube.

On the contrary, in the case where the present furnace was used, the inner surface of the inner cylinder 18 was slightly oxidized after the thermal treatments, but the SiC coating of the muffle tube was not changed at all. The pure silica single mode optical fiber drawn from the thermally treated preform with the present furnace had very low average transmission loss of 0.173 dB/km at a wavelength of 1.55 μm.

In the case where the glass preform was thermally treated with using the present furnace comprising the inner tube made of highly pure carbon, air may enter the muffle tube when the porous glass preform 15 is inserted in the inner tube, whereby the inner tube may be deteriorated due to oxidation. In order to prevent such oxidation, the insertion of the glass preform may be performed at a temperature below 400 °C or an upper portion of the muffle tube 11 may be partitioned as a front chamber.

According to the present furnace for the production of the highly pure quartz preform by the thermal treatment, a small amount of oxygen and water contained in the glass preform firstly reacts with the inner cylinder during the thermal treatment of the preform since the inner cylinder is detachably fitted inside the muffle tube. Therefore, oxygen and water do not directly deteriorate the muffle tube, whereby the life of the muffle tube is extended and the preform is suitably thermally treated.

In the second aspect of the present invention, there is provided a furnace as described in the first aspect of the present invention, wherein at least one highly pure carbon made member which constitutes the muffle tube or the inner cylinder, or optional additional element in the muffle tube which is necessary for the muffle tube (for example, a part of supporting rod) is made of a graphite material selected from the group consisting of a graphite material which is produced by self-sintering mesophase powder and a highly

pure graphite material consisting of raw particles selected from the group consisting of particles having a maximum particle size of not larger than 50 µm and particles having an average particle size of not larger than 20 µm.

In the furnace of the present invention, the muffle tube may comprise of a single highly pure carbon made member or a plurality of the highly pure carbon made members. When the muffle tube comprises the single highly pure carbon made member, the member may be made of either of the graphite materials as described above. When the muffle tube consists of a plurality of the highly pure carbon made members, at least one member which is heated to the highest temperature (for example 1200 °C) is preferably made of either of the graphite materials as described above. All of the members may be made of such graphite materials.

Though any member may be provided with the gas impermeable coating made of, for example, SiC, pyrolitic carbon or vitreous carbon, it is preferable to provide at least the member which is heated to such highest temperature with the coating.

In the case where the quartz glass preform is thermally treated in the present furnace having the muffle tube, the inner tube and optional element (which is, hereinafter, referred to as also muffle tube assembly) at least a part of which is made of the graphite material produced by self-sintering the mesophase powder, generation of the carbon powder is hardly observed when the muffle tube assembly is consumed by a trace amount of water and oxygen, whereby no bubble is generated in the quartz glass preform because of absence of carbon powder. Thus, there is no strength reduction of the optical fiber which is produced from the preform.

The term "graphite material produced by self-sintering the mesophase powder" is intended to mean a graphite material produced by self-sintering an intermediate liquid phase in which fine spherical particles having optical anisotropy are generated during an initial period of a thermal treatment of coal or heavy oil. Such particles are called mesophase fine spherical particles. The graphite material which is produced by self-sintering the mesophase powder is commercially available from Sumitomo Metal Industries, Ltd. (Osaka, Japan) and Toshiba Ceramics Co., Ltd. (Tokyo, Japan).

In the case where the quartz glass preform is thermally treated in the present furnace having the muffle tube assembly comprising (at least one member made of) the highly pure graphite material consisting of the raw particles having a maximum particle size (diameter) of not larger than 50 µm or an average particle size (diameter) of not larger than 20 µm, generation of the carbon powder is hardly observed. Even when the carbon powder is generated, such graphite powder is so fine that it reacts with SiO_2 on the preform surface to produce gases of SiO and CO, whereby no bubble is generated because of the absence of the carbon powder in the preform. Thus, there is no strength reduction of the optical fiber which is produced from the preform.

The furnace of the preferred embodiment of the second aspect of the present invention is schematically shown also in Fig. 2 as a sectional view. In this, the muffle tube 11 comprises three members, that is the upper member 34, the middle member 35 and the lower member 36, and the inner cylinder 18 which is made of the graphite material produced by self-sintering the mesophase powder. The inner cylinder may be provided with the gas impermeable coating (not shown).

The middle member 35 is preferably made of the graphite material produced by self-sintering the mesophase powder provided with the gas impermeable coating (not shown).

Under the condition such that the gas impermeable coating is not so consumed or the coating withstands the deterioration for a sufficient period, the middle member 35 is not necessarily made of the graphite material produced by self-sintering the mesophase powder. Other members such as the upper member and the lower member may be optionally made of such graphite material and may be provided with the gas impermeable coating.

In the construction as shown in Fig. 2, the inner cylinder may be made of the highly pure graphite material consisting of the raw particles having a maximum particle size of not larger than 50 µm or an average particle size of not larger than 20 µm.

When a small amount of water or oxygen consumes the muffle tube material so that the carbon powder is generated during the thermal treatment of the preform, the particles of the carbon powder are extremely fine.

In this case, even when the carbon powder penetrates the preform to a slightly inner portion, it is completely gasified because of following reaction between the carbon powder and quartz:

$SiO_2 + C \rightarrow SiO\ (gas) + CO\ (gas)$

Thus, no bubble is generated in the preform and the strength reduction of the optical fiber drawn from the preform is prevented.

In this embodiment, the middle member 35 is also preferably made of the highly pure graphite material

coated with the gas impermeable layer as described above.

Under the condition such that the gas impermeable coating is not so consumed or the coating withstands the deterioration for a sufficient period, the middle member 35 is not necessarily made of such highly pure graphite material. Other members such as the upper member and the lower member may be optionally made of such graphite material and provided with the gas impermeable coating.

Example 2

The furnace as shown in Fig. 2 was used. The inner cylinder 18 was made of the graphite material produced by self-sintering the mesophase powder. A bulk specific gravity of the graphite material was 1.95 and a maximum size of the raw particles for the graphite material was 5 µm. The middle member 35 of the muffle tube 3 was made of a usual isotropical graphite and was coated with the gas impermeable pyrolysis carbon. The upper and lower members 34 and 36 were made of usual isotropical graphite and were coated with the gas impermeable SiC.

Eighty preforms produced by VAD method were sintered in the furnace under conditions shown in following Table 2:

Table 2

Treatment	Temperature	Atmosphere gas
Dehydration	950-1100 °C	He, $SiCl_4$ or Cl_2
Sintering	1550-1650 °C	He

During the sintering treatment, no generation of the carbon powder was observed in the muffle tube and no bubble was observed in the surface layer portion of the sintered preform.

An optical fiber was drawn from each of the tenth and the eightieth preforms. Twenty sample fibers each having a length of 20 m were cut from each optical fiber and they were tested on the longitudinal tensile strength. There was no sample fiber which had a small strength, that is, a tensile strength of less than 4.5 kg-f.

Comparative Example 1

For comparison with the above example, Example 1 was repeated except that a furnace which comprised an inner cylinder made of a usual isotropical carbon material was used. The carbon material had a bulk specific gravity of 1.77 and a maximum raw particle size of 60 µm. Sixty preforms were sintered under the same conditions as in Example 1.

An optical fiber was drawn from each of the tenth and the sixtieth preforms and similarly the longitudinal tensile test was performed.

There is no sample fiber drawn from the tenth preform which had the small strength. However, 30 % of the sample fibers drawn from the sixtieth preform had the small strength. When the sixtieth preform was treated, the carbon powder was generated in the muffle tube and the bubbles were observed in the preform surface portion.

As described above, the furnace of the present invention having the muffle tube assembly at least a part of which is made of the graphite material produced by self-sintering the mesophase powder can be used to thermally treat the preform for a long period without the generation of the carbon powder in the muffle tube and the strength reduction of the optical fiber drawn from the preform.

Example 3 and Comparative Example 2

The furnace as shown in Fig. 2 was used. The inner cylinder 8 was made of the graphite material as shown in Table 3. In Table 3, Samples A and B are for Example 3 and Samples C and D are for Comparative Example 2. Eighty preforms produced by VAD method were thermally treated under the same

conditions as in Example 2.

Table 3

Sample	Material	Shaping method	Bulk specific gravity	Maximum particle size	Average particle size
A	Isotropical graphite	Rubber pressing	1.84	20 µm	-
B	Isotropical graphite	Rubber pressing	1.85	-	10 µm
C	Isotropical graphite	Rubber pressing	1.77	60 µm	-
D	Anisotropical graphite	Extrusion	1.64	250 µm	-

An optical fiber was drawn from each of the tenth and the eightieth preforms. twenty sample fibers each having a length of 20 m were cut from each fiber and tested on the longitudinal tensile strength. From the test data, a ratio of the optical fiber having the small strength, that is, the optical fiber having the 4.5 kg-f was calculated.

The results are shown as follows:

Sample A

Between the fiftieth and the sixtieth preforms, extremely fine carbon powder was generated in the muffle tube. No bubble was observed in the surface layer even in the eightieth sintered preform.

The results of the longitudinal tensile test were such that 5 % of the sample fibers drawn from the tenth preform had the small strength, and no sample fiber drawn from the eightieth preform had the small strength.

Sample B

Between the fiftieth and the sixtieth preforms, extremely fine carbon powder was generated in the muffle tube. No bubble was observed in the surface layer even in the eightieth sintered preform.

The results of the longitudinal tensile test were such that no sample fiber drawn from the tenth preform had the small strength, and 5 % of the sample fibers drawn from the eightieth preform had the small strength.

Sample C

Between the fiftieth and the sixtieth preforms, slightly large carbon powder was generated in the muffle tube.

Because of the generation of the bubbles in the surface layer, the thermal treatment was stopped at the sixtieth preform. Optical fibers were drawn from the sixtieth preform and the longitudinal tensile test was performed. The ratio of the sample fibers having the small strength was 30 %.

No sample fiber drawn from the tenth preform had the small strength.

Sample D

Between the fortieth and the fiftieth preforms, slightly large carbon powder was generated in the muffle tube. Since bubbles were generated in the surface layer of the preform, the thermal treatment was stopped at the fiftieth preform. Optical fibers were drawn from the fiftieth preform and the longitudinal tensile test was performed. The ratio of the sample fibers having the small strength was 35 %.

Five percents of the sample fibers drawn from the tenth preform had the small strength.

As described above, the furnace of the present invention having the muffle tube assembly at least one

member of which is made of the highly pure graphite consisting of the raw particles having a maximum size of not larger than 50 μm or an average size of not larger than 20 μm can be used to thermally treat the preform for a long period without the generation of the carbon powder in the preform and the strength reduction of the optical fiber drawn from the preform.

Claims

1. A furnace comprising a muffle tube made of a gas impermeable and heat resistant material, a heater in a furnace body which surrounds the muffle tube and an inner cylinder made of a heat resistant material which is detachably fitted inside the muffle tube near the heater for a thermal treatment of a quartz glass preform by inserting the preform in the muffle tube, wherein said muffle tube comprises a material selected from a group consisting of highly pure carbon coated with gas impermeable silicon carbide, sintered silicon carbide coated with gas impermeable silicon carbide, gas impermeable silicon carbide and highly pure carbon coated with gas impermeable carbon, and said inner cylinder comprises a material selected from a group consisting of highly pure carbon, highly pure carbon coated with gas impermeable silicon carbide and highly pure carbon coated with gas impermeable carbon.

2. The apparatus according to claim 1, wherein at least one of the highly pure carbon made members which constitute the muffle tube, the inner cylinder and an optional element necessary for the muffle tube is made of a graphite material which is produced by self-sintering mesophase powder.

3. The apparatus according to claim 1, wherein at least one of the highly pure carbon made members which constitute the muffle tube, the inner cylinder and an optional element necessary for the muffle tube is made of a highly pure graphite material consisting of raw particles selected from the group consisting of particles having a maximum particle size of not larger than 50 μm and particles having an average particle size of not larger than 20 μm.

Fig. 1

Prior Art

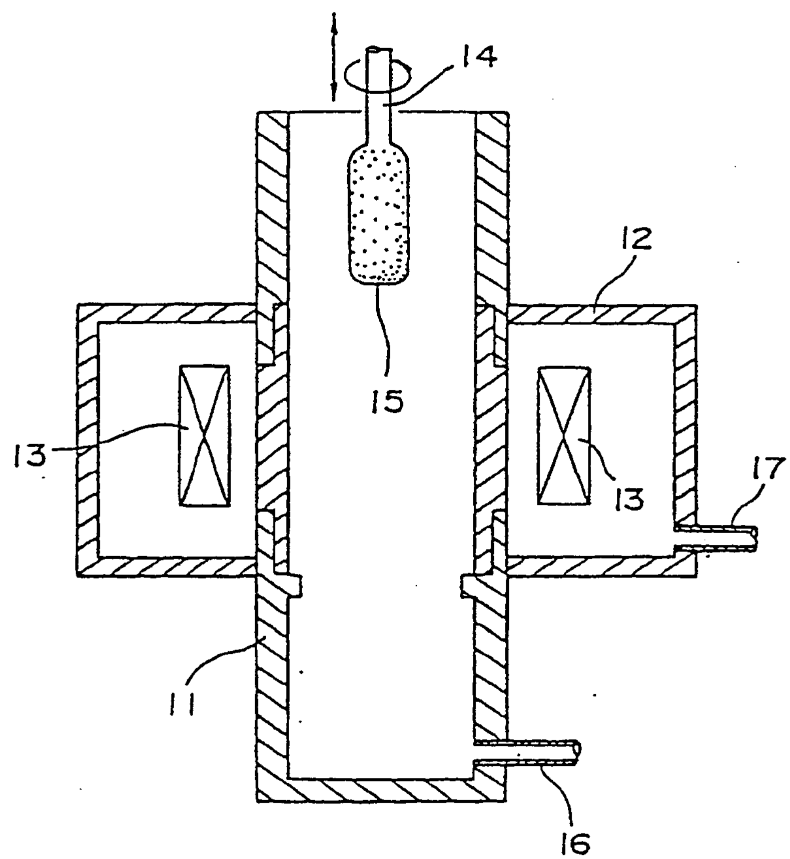
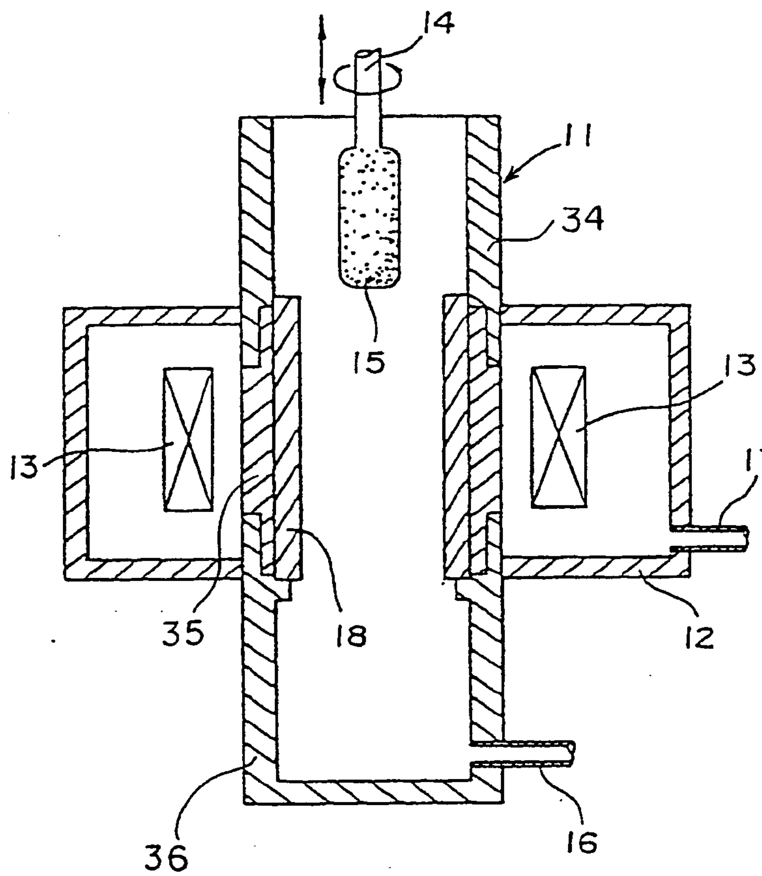


Fig. 2



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EUROPEAN SEARCH REPORT

Application Number

EP 90 11 8393

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
P,X	EP-A-0 380 054 (SUMITOMO ELECTRIC INDUSTRIES LTD) * Claims; figures 3,4; page 5, lines 14-21 *	1	C 03 B 37/014
A	EP-A-0 302 121 (SUMITOMO ELECTRIC INDUSTRIES LTD) * Claims 1-3,9,10; figures 3-5 *	1	
A	DE-A-2 818 550 (SUMITOMO ELECTRIC INDUSTRIES LTD) * Claim 4 *	2	
A	PATENT ABSTRACTS OF JAPAN, vol. 11, no. 53 (C-404)[2500], 19th February 1987; & JP-A-61 215 226 (FURUKAWA ELECTRIC CO., LTD) 25-09-1986 * Figures 1,3 *	1	

TECHNICAL FIELDS SEARCHED (Int. Cl.5)

C 03 B 37/014

The present search report has been drawn up for all claims

Place of search	Date of completion of search	Examiner
The Hague	07 December 90	STROUD J.G.

CATEGORY OF CITED DOCUMENTS

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